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bp



CERTIFIED MAIL
RETURN RECEIPT REQUESTED

December 13, 2011

Indiana Department of Environmental Management OWO Data Management Section 100 North Senate Avenue Indianapolis, IN 46206 BP Products North America Inc. 2815 Indianapolis Blvd. P.O. Box 710 Whiting, IN 46394-0710 USA

Subject: NPDES Permit No. 0000108, Semi Annual WET Testing Results

Please find enclosed two copies of the Whole Effluent Toxicity Report for BP Products North America Inc. – Whiting Business Unit for the month of October 2011. Results are reported according to EPA 821-R-02-013 Section 10 (Report Preparation) for NPDES permit IN0000108 Outfall 005 Effluent. Chronic Toxicity TUc was 2.0 and Acute Toxicity TUa was <1.0.

BP plans to conduct WET testing in April and October of each calendar year. The next sampling event will be in April 2012.

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

If you have any questions or need any additional information, please contact Tim Chen at (219) 473-1286.

Sincerely,

Nick Speneer

Business Unit Leader

**Attachments** 

CC: Nick Ream (IDEM - Merrillville, IN)



# Whole Effluent Toxicity Test Results

Prepared for:

BP Products North America Whiting, Indiana

Prepared by: ENVIRON International Corporation Nashville, Tennessee

Date: October 2011

Project Number: 20-19696D

## ENVIRON

November 1, 2011

Mr. Rick Solan BP Products North America 2831 Indianapolis Blvd., Stop 10-2 Whiting, IN 46394

Re:

Whole Effluent Toxicity Test Results – October 2011 ENVIRON Project No. 20-19696D

Dear Mr. Solan:

Attached are the results of the *Pimephales promelas* (fathead minnow) chronic (7-day) Whole Effluent Toxicity (WET) test performed with composite samples of Outfall 005 effluent. This cover letter contains a test overview and summary of test results. The detailed report formatted to meet guidelines specified in your NPDES discharge permit (i.e., following the outline in Section 10 of EPA 821-R-02-013) is attached.

Three, 24-hour composite samples were evaluated in the WET test. Testing was conducted in accordance with Permit No. IN0000108. Samples were collected on October 10, 12, and 14, 2011 and used at the ENVIRON Toxicology Laboratory within 36 hours of collection at temperatures meeting the USEPA-required receipt temperature range of 0 to 6.0 °C (see chain-of-custody forms). Test organisms were exposed to effluent concentrations of 6.25, 12.5, 25, 50, and 100 percent effluent and a moderately hard water control. Chronic toxicity test methods followed EPA 821-R-02-013, Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, Fourth Edition Section 11. Fish were fed twice daily with 0.15 g artemia (brine shrimp nauplii) that was rinsed with freshwater to remove salinity before use. Test results are presented below:

| Chronic Test Results – Ou | ıtfali 005 Effluent |
|---------------------------|---------------------|
| 96-hr LC50                | > 100%              |
| TUa (100/LC50)            | < 1.0               |
| NOEC Survival (7 day)     | 50%                 |
| NOEC Growth (7 day)       | 50%                 |
| IC25 (7 day)              | 68%                 |
| TUc (100/NOECgrowth)      | 2.0                 |

No acute (96 hour) toxicity was observed (LC50 greater than 100 percent effluent). Chronic (seven day) toxicity was observed in the 100 percent effluent exposures as indicated by survival and growth NOEC (No Observed Effects Concentration) values of 50 percent effluent. This corresponds to an NOEC-based TUc value of 2.0. The chronic 25 percent Inhibition Concentration (IC25) value was 68 percent effluent.

201 Summit View Drive, Suite 300, Brentwood, TN 37027 Tel: +1 615.377.4775 Fax: +1 615.377.4976

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Lab Certifications: AR (#02-008-0), AZ, CA (#2465), FL (#E87896), IA (#386), KS, KY, LA (#02061), MI, NC, OK (#9973), SC (#84015), T104704410-08-TX, VA (#460171), WI (#399050850), WV
Test Results Contained in this Report Meet NELAP Requirements
ENVIRON Test Log No. 13888

Test controls met USEPA criteria for test acceptability. The concentration-response relationship for growth is described as Number 3 in EPA821-B-00-004 Method Guidance and Recommendations for Whole Effluent Toxicity (WET) Testing. The dose-response indicates stimulation at lower concentrations, and significant effect at highest concentrations. Test precision as measured by the Percent Minimum Significant Difference (PMSD) value for this test was 24.1 percent, which is within the USEPA PMSD bounds of 12 to 30 percent for fathead minnow growth. This test is valid for determination of permit compliance. The monthly reference toxicant test for fathead minnow met all test acceptability criteria and was in acceptable range for determining normal test performance.

In accordance with NELAP requirements for listing the number of report pages, this report contains 32 pages, which include the cover letter, detailed report (Attachment 1), associated chemical data (Attachment 2), statistical analyses and raw data (Attachment 3), chain-ofcustody forms (Attachment 4), reference toxicant data (Attachment 5), and associated separator pages.

Thank you for the continued opportunity to be of service to BP Products North America. If you have any questions concerning these data, please call Teri Horsley at (615) 377-4775, ext 168.

Sincerely,

Mr. Rick Solan

**ENVIRON International Corporation** 

Teri L. Horsley

cc: Rose Hererra

Jew L Houly

Project Manager, Ecotoxicology

Robin L. Garibay, REM

Rober J. Havi bay

Principal

Attachment 1: Detailed Report

20-19696D € N V I R O N

### ENVIRON

# BP Whiting Detailed Toxicity Test Report – October 2011 Fathead Minnow Chronic Test

#### 1 Introduction

The BP Whiting Refinery is located in Whiting, Indiana at 2815 Indianapolis Boulevard and discharges treated effluent to Lake Michigan under a National Pollutant Discharge Elimination System (NPDES) permit (Permit No. IN0000108) as administered by the Indiana Department of Environmental Management (IDEM). The subject discharge permit requires semi-annual (twice per year) Whole Effluent Toxicity (WET) testing with the fathead minnow. In support of these discharge monitoring requirements, the WET tests described herein were conducted. Testing was performed by:

ENVIRON International Corporation (ENVIRON) 201 Summit View Drive Lower/Lab Level Brentwood, TN 37027 (615 377-4775)

The objective of this test was to provide WET test data in support of BP Whiting's NPDES discharge monitoring requirements for Outfall 005.

#### 2 Plant Operations

The BP Whiting Refinery produces various grades of gasoline, diesel and heating fuel, asphalt, and coke, among other products from refined crude oil. The facility operates 24 hours a day, seven days per week under normal operations. Some facility processes are occasionally suspended for maintenance or as a result of unplanned events (e.g., equipment failure, etc.). Wastewater treatment consists of bar screening, grit removal, oil/water separator, storm surge tank, equalization tank, flocculation/flotation, activated sludge, settling and multimedia filtration prior to discharge to Lake Michigan. Wastewater retention time is approximately 17 to 18 hours for this approximately average of 20 MGD discharge through Outfall 005. The design flow of the treatment plant at the time of WET test sampling was 35 MGD.

#### 3 Effluent and Dilution Water

#### 3.1 Effluent Samples

Composite Outfall 005 effluent samples were collected from the lake front sample shed at the NPDES permit-specified sample location for WET and chemical sampling of this outfall. Composite samples were obtained from a continuous flow of effluent pulled from the effluent discharge to provide representative effluent samples. The latitude and longitude of this sampling point is 41° 40′ 36″ N and 87° 28′ 16″ W. Three effluent samples were collected on the following dates (date indicates the day on which the composite sample was completed): October 10, 12, and 14, 2011. The composite sampler initiated sampling at 0751 for the first sample and 0700 for the second and third samples and sampled hourly for 24 hours on the dates indicated, providing the permit-specified 24 individual sample aliquots (within a 24 hour period) composited for toxicity testing. An Isco automatic sampler was used to collect the hourly samples that were composited into a common sample container (maintained on ice during collection). WET testing was supported by chemical analyses

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(Attachment 2). The physical and chemical data associated with each sample used in WET testing are provided on the laboratory bench sheets documenting these and other sample conditions (Attachment 3). The mean daily discharges on sample collection dates were 12.6 mgd, 14.5 mgd, and 14.1 mgd, on October 10, 12, and 14, 2011, respectively. The lapsed time between sample collection and receipt at the ENVIRON WET testing laboratory was 23 hours and 54 minutes, 24 hours and 26 minutes, and 25 hours and 42 minutes, for samples received on October 11, 13, and 15, respectively. The respective sample receipt temperatures were 0.4 °C, 0.1 °C, and 0.6 °C.

#### 3.2 Dilution Water

The dilution and control water for this test was USEPA moderately hard water, prepared in accordance with EPA 821-R-02-013. The water was prepared by ENVIRON using deionized water to which the four reagent-grade salts specified by USEPA were added and aerated for a minimum of 24 hours before use. No pre-treatment of the water occurred following this preparation. As detailed in Attachment 3, dilution water hardness and alkalinity ranged from 84 to 86 mg/L CaCO<sub>3</sub> and from 50 to 52 mg/L CaCO<sub>3</sub>, respectively. Control water pH ranged from approximately 7.2 to 7.9 s.u., and dissolved oxygen was always greater than 8 mg/L during the test.

#### 4 Test Method

The fathead minnow chronic WET test method detailed in Section 1000.0 of EPA 821-R-02-013 (Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms – Fourth Edition) was followed. Per the method, sevenday fathead minnow survival and growth (dry weight), were the test endpoints assessed. There were no deviations from this test method. The test was initiated at 1030 on October 11<sup>th</sup> and terminated at 1230 on October 18<sup>th</sup>. Disposable plastic 400 mL test vessels containing 350 mL test solutions were used. Four replicate exposures of 10 organisms were used for each of the five effluent exposures and the control exposures. Test organisms had been acclimated to the control water at 24 °C prior to test initiation. Test temperatures ranged from 24.0 to 25.1 °C. Neither aeration nor pH adjustment were required during the test. Test organisms were fed a minimum of 0.15 g of live Artemia (brine shrimp) nauplii twice daily. Brine shrimp were rinsed with freshwater to remove salinity prior to use in tests. Test solutions were renewed daily.

#### 5 Test Organisms

Fathead minnows (Pimephales promelas) less than 24 hours old at test initiation were used in this testing. Organisms were obtained from a commercial laboratory (Environmental Consulting and Testing) with whom ENVIRON has a long-standing record of successful use in WET tests. Organisms were obtained from laboratory-reared stock traceable to USEPA cultures. Taxonomic verification is provided by the laboratory. No treatments for disease were used on the fish used in these WET tests.

#### 6 Quality Assurance

Reagent grade sodium chloride (NaCl, Reagents, Inc.) is used in monthly reference toxicant tests conducted to document test organism sensitivity and test endpoint precision. Organisms tested are from the same commercial source used in WET tests, and organism handling and testing in reference toxicant tests is identical to that of WET tests. At the time

of this test, the most recently completed reference toxicant test was initiated on October 4, 2011. This test was found to be within method-specified control bounds as indicated in Attachment 5 where the control chart is provided. USEPA moderately hard water was the dilution and control water in all reference toxicant tests. The 25 percent inhibition concentration is used to track reference toxicant test performance and as such the percent minimum significant difference statistic is not applicable. As documented in ENVIRON's Standard Operating Procedures (SOP) manual, pH, dissolved oxygen, and conductivity meters calibrated daily according to manufacturer's instructions were used to document these water quality conditions during reference toxicant tests. Standard, titration-based methods were used to document control water hardness and alkalinity as specified in the ENVIRON SOP manual.

#### 7 Results

Raw WET test data are provided in Attachment 3, serving as documentation of the daily effects observed in each test and control replicate. Final test results are also provided in graphical form in Attachment 3 for the specified biomass basis (i.e., integration of survival and growth endpoints). Commercial software (ToxCalc Version 5.0), which is designed specifically to meet USEPA-specified statistical requirements outlined in Section 9.0 of EPA 821-R-02-013 was used for analysis of the fathead minnow survival and growth data. All of the physical and chemical data associated with the toxicity tests are listed in the test bench sheets provided in Attachment 3. In summary, these are:

| Parameter (units)                    | Range observed in Test<br>(all exposures) |
|--------------------------------------|---|
| pH (s.u.)                            | 6.94 to 7.84                              |
| Dissolved Oxygen (mg/L)              | 7.4 to 8.8                                |
| Conductivity (umno/cm)               | 377 to 3,230                              |
| Temperature (°C)                     | 24.0 to 25.1                              |
| Alkalinity (mg/L CaCO <sub>3</sub> ) | 75 to 160 (100% effluent)                 |
| Hardness (mg/L CaCO <sub>3</sub> )   | 212 to 232 (100% effluent)                |

The acute (96 hour) median lethal concentration (LC50) value for this test is greater than 100 percent effluent based on the absence of 50 percent or more morality in all test exposures (five percent mortality was observed in the 100 percent effluent exposure). This corresponds to an acute toxicity unit (TUa) value of less than 1. The biomass method-based chronic tests endpoints of a 25 percent inhibition concentration (IC25) and No Observed Effects Concentration (NOEC) value based on survival and growth were assessed. The respective IC25 and NOEC values for this test were 68 percent and 50 percent effluent. The resulting NOEC-based chronic toxic unit value (TUc) is 2.0. The Percent Minimum Significant Difference (PMSD) value (applicable to the NOEC value only) for this test was 24.1 percent.

Attachment 4 contains chain-of-custody documentation.

#### 8 Conclusions and Recommendations

There are no WET permit limits in the current NPDES discharge permit. Only twice per year monitoring is required. No acute toxicity was indicated during this test; however, chronic toxicity to full-strength effluent was indicated.

**Attachment 2:** Chemical Data

20-19696D € N V I R O N

| ENVIRON . |                              |              |                       |        |           | Oct-2011   |          |        |           |            |          |        |  |            |          |
|-----------|------------------------------|--------------|-----------------------|--------|-----------|------------|----------|--------|-----------|------------|----------|--------|--|------------|----------|
| Test Log  |                              |              |                       |        | Sun       | day        |          |        | Tue       | sday       |          |        | Thur   | sday       |          |
|           |                              |              |                       |        | 10/9/     | 2011       |          |        | 10/11     | /2011      |          |        | 10/13  | /2011      |          |
| ĕι        | IMS ID                       |              |                       |        | 1840792/  | 1841137    |          |        | 1841642   | /1842111   |          |        | 1842648  | 1843092    |          |
| ည္ထဲ      |                              | Units of     | Analysis              | •      |           | Sample     |          |        |           | Sample     |          |        |  | Sample     |          |
| ₩ F       | Parameter                    | Measure      | Method                | Result | Comp/Grab | Date       | Tech     | Result | Comp/Grab | Date       | Tech     | Result | Comp/Grab  | Date       | Tech     |
| _         | BOD5                         | mg/L         | SM 5210 B             | 1.6    | Comp      | 10/9/2011  | NP       | 2.6    | Comp      | 10/11/2011 | JPO      | 8.3    | Comp   | 10/13/2011 | JPO      |
| ī         | SS                           | mg/L         | SM 2540 D             | 9.2    | Comp      | 10/9/2011  | NP       | 13.2   | Comp      | 10/11/2011 | NP       | 11.6   | Comp   | 10/13/2011 | NP       |
| Ī         | COD                          | mg/L         | SM 5220 D             | 51     | Comp      | 10/9/2011  | NP       | 34     | Comp      | 10/11/2011 | NP       | 60     | Comp   | 10/13/2011 | NP       |
| Ī         | Oil & Grease                 | mg/L         | EPA 1664A             | <0.3   | Grab      | 10/10/2011 | NP       | 0.5    | Grab      | 10/12/2011 | NP       | 1.0    | Grab   | 10/14/2011 | JPO      |
| 7         | Ammonia                      | mg/L         | SM 4500 NH3 F         | <0.10  | Comp      | 10/9/2011  | JPO      | <0.10  | Comp      | 10/11/2011 | JPO      | 0.33   | Comp   | 10/13/2011 | JPO      |
| ħ         | Total Chromium               | mg/L         | SM 3111 B             | <0.01  | Comp      | 10/9/2011  | JPO      | <0.01  | Comp      | 10/11/2011 | JPO      | <0.01  | Comp   | 10/13/2011 | JPO      |
| F         | lexavalent Cr                | mg/L         | SM 3500 Cr D          | <0.005 | Grab      | 10/10/2011 | NP       | <0.005 | Grab      | 10/12/2011 | NP       | <0.005 | Grab   | 10/14/2011 | NP       |
| F         | Phenolics                    | mg/L         | SM 5530 D / EPA 420.1 | <0.01  | Comp      | 10/9/2011  | JPO      | <0.01  | Comp      | 10/11/2011 | JPO      | <0.01  | Comp   | 10/13/2011 | JPO      |
| ð F       | Phospsorous (PO4)            | mg/L         | SM 4500 P             | 0.29   | Comp      | 10/9/2011  | NP       | 0.10   | Comp      | 10/11/2011 | NP       | 0.02   | Comp   | 10/13/2011 | NP       |
| 0 -       | Sulfides                     | mg S2-/L     | SM 4500 S2- D         | 0.03   | Comp      | 10/9/2011  | JPO      | 0.02   | Comp      | 10/11/2011 | JPO      | 0.02   | Comp   | 10/13/2011 | NP       |
| ~         | oH                           | S.U.         | SM 4500 H+ B          | 7.2    | Grab      | 10/10/2011 | PG       | 7.10   | Grab      | 10/12/2011 | GC       | 6.8    | Grab   | 10/14/2011 | П        |
| ľ         |                              |              |                       |        |           |            |          |        |           |            |          |        |  |            |          |
|           |                              |              |                       |        |           |            |          |        |           |            |          |        |  |            |          |
| 1         | Microbac Tests               |              |                       |        |           |            |          |        |           |            |          |        | <del>                                     </del> |            |          |
| -         | Fotol Maraun                 | 20/1         | 1631 E                | 2.83   | Grab      | 10/10/2011 | Microbac | 5.88   | Grab      | 10/12/2011 | Microbac | 11.0   | Grab   | 10/14/2011 | Microbac |
| ŀ         | Total Mercury Total Vanadium | ng/L<br>mg/L | 200.8_R5.4            | 0.033  | Comp      | 10/9/2011  | Microbac | 0.018  | Comp      |            | Microbac |        | Comp   |            | Microbac |

Attachment 3: Statistical Anaylses and Raw Data

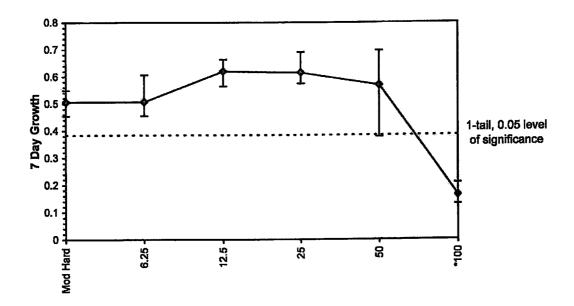
20-19696D E N V I R O N

|   |                          |        | La                  | rval Fish G      | owth and Surviva | al Test-7 Day G            | rowth   |
|---|--------------------------|--------|---------------------|------------------|------------------|----------------------------|---|
| Start Date:<br>End Date:<br>Sample Date:<br>Comments: | 10/11/2011<br>10/18/2011 |        | Test ID:<br>Lab ID: | 13888<br>ENVIRON |                  | Sample ID:<br>Sample Type: | BP_001005 राज क्षिणी<br>EFF2-Industrial<br>PP-Pimephales promelas |
| Conc-%  | 1                        | 2      | 3                   | 4                |                  |                            |   |
| Mod Hard  | 0.5150                   | 0.4550 | 0.5060              | 0.5490           |                  |                            |   |
| 6.25  | 0.4560                   | 0.4960 | 0.4710              | 0.6070           |                  |                            |   |
| 12.5  | 0.6040                   | 0.6480 | 0.5640              | 0.6630           |                  |                            |   |
| 25  | 0.5970                   | 0.6900 | 0.5740              | 0.5970           |                  |                            |   |
| 50  | 0.6960                   | 0.6320 | 0.5640              | 0.3780           |                  |                            |   |
| 100   | 0.1450                   | 0.1300 | 0.1650              | 0.2080           |                  |                            |   |

|          |        | Moon   |        |        | Transform | n: Untran | sformed |        |          | 1-Tailed |  |
|----------|--------|--------|--------|--------|-----------|-----------|---------|--------|----------|----------|--|
| Conc-%   | Mean   | N-Mean | Mean   | Min    | Max       | CV%       | N       | t-Stat | Critical | MSD      |  |
| Mod Hard | 0.5062 | 1.0000 | 0.5062 | 0.4550 | 0.5490    | 7.676     | 4       |        |          |          |  |
| 6.25     | 0.5075 | 1.0025 | 0.5075 | 0.4560 | 0.6070    | 13.469    | 4       | -0.025 | 2.410    | 0.1222   |  |
| 12.5     | 0.6197 | 1.2242 | 0.6197 | 0.5640 | 0.6630    | 7.231     | 4       | -2.238 | 2.410    | 0.1222   |  |
| 25       | 0.6145 | 1.2138 | 0.6145 | 0.5740 | 0.6900    | 8.379     | 4       | -2.135 | 2.410    | 0.1222   |  |
| 50       | 0.5675 |        | 0.5675 | 0.3780 | 0.6960    | 24.203    | 4       | -1.208 | 2.410    | 0.1222   |  |
| *100     | 0.1620 | 0.3200 | 0.1620 | 0.1300 | 0.2080    | 20.897    | 4       | 6.789  | 2.410    | 0.1222   |  |

| Auxiliary Tests                     |              |            | · · · · · · · · · · · · · · · · · · · |     | Statistic |         | Critical |         | Skew    | Kurt    |
|-------------------------------------|--------------|------------|---------------------------------------|-----|-----------|---------|----------|---------|---------|---------|
| Shapiro-Wilk's Test indicates nor   | mal distribu | ition (p > | 0.05)                                 | - * | 0.93043   |         | 0.916    |         | -0.6193 | 2.67151 |
| Bartlett's Test indicates equal var |              |            | ,                                     |     | 8.29943   |         | 15.0863  |         |         |         |
| Hypothesis Test (1-tail, 0.05)      | NOEC         | LOEC       | ChV                                   | TU  | MSDu      | MSDp    | MSB      | MSE     | F-Prob  | df      |
| Dunnett's Test                      | 50           | 100        | 70.7107                               | 2   | 0.1222    | 0.24138 | 0.11701  | 0.00514 | 3.4E-07 | 5, 18   |
| Treatments vs Mod Hard              |              |            |                                       |     |           |         |          |         |         |         |

#### Dose-Response Plot



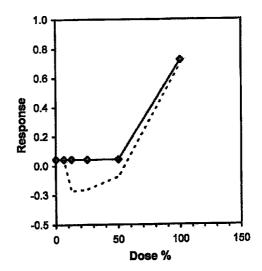
|   |                          |        | Lai                 | val Fish Gr      | owth and Surviv | il Test-7 Day Bio          | omass  |
|---|--------------------------|--------|---------------------|------------------|-----------------|----------------------------|--|
| Start Date:<br>End Date:<br>Sample Date:<br>Comments: | 10/11/2011<br>10/18/2011 |        | Test ID:<br>Lab ID: | 13888<br>ENVIRON | EPA/821/R-02-01 | Sample ID:<br>Sample Type: | BP_001 005 Tut 12-11/11 EFF2-Industrial PP-Pimephales promelas |
| Conc-%  | 1                        | 2      | 3                   | 4                |                 |                            |  |
| Mod Hard  | 0.5150                   | 0.4550 | 0.5060              | 0.5490           |                 |                            |  |
| 6.25  | 0.4560                   | 0.4960 | 0.4710              | 0.6070           |                 |                            |  |
| 12.5  | 0.6040                   | 0.6480 | 0.5640              | 0.6630           |                 |                            |  |
| 25  | 0.5970                   | 0.6900 | 0.5740              | 0.5970           |                 |                            |  |
| 50  | 0.6960                   | 0.6320 | 0.5640              | 0.3780           |                 |                            |  |
| 100   | 0.1450                   | 0.1300 | 0.1650              | 0.2080           |                 |                            |  |

|          |        |        |        | Transforn | n: Untrans | sformed | Isoto |        |        |
|----------|--------|--------|--------|-----------|------------|---------|-------|--------|--------|
| Conc-%   | Mean   | N-Mean | Mean   | Min       | Max        | CV%     | N     |        | N-Mean |
| Mod Hard | 0.5062 | 1,0000 | 0.5062 | 0.4550    | 0.5490     | 7.676   | 4     | 0.5631 | 1.0000 |
| 6.25     | 0.5075 | 1.0025 | 0.5075 | 0.4560    | 0.6070     | 13.469  | 4     | 0.5631 | 1.0000 |
| 12.5     | 0.6197 | 1.2242 | 0.6197 | 0.5640    | 0.6630     | 7.231   | 4     | 0.5631 | 1.0000 |
| 25       | 0.6145 | 1.2138 | 0.6145 | 0.5740    | 0.6900     | 8.379   | 4     | 0.5631 | 1.0000 |
| 50       | 0.5675 | 1.1210 | 0.5675 | 0.3780    | 0.6960     | 24.203  | 4     | 0.5631 | 1.0000 |
| 100      | 0.1620 | 0.3200 | 0.1620 | 0.1300    | 0.2080     | 20.897  | 4     | 0.1620 | 0.2877 |

| Auxiliary Tests  | Statistic | Critical | Skew Kurt       |
|--|-----------|----------|-----------------|
| Shapiro-Wilk's Test indicates normal distribution (p > 0.05) | 0.93043   | 0.916    | -0.6193 2.67151 |
| Bartlett's Test indicates equal variances (p = 0.14)         | 8.29943   | 15.0863  |                 |

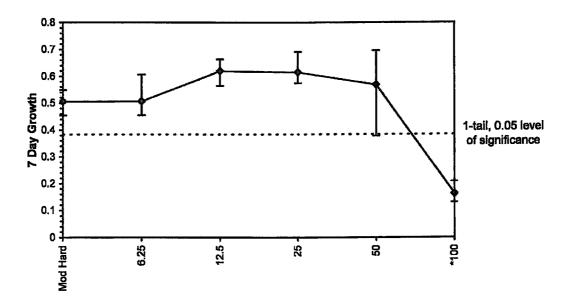
Linear Interpolation (200 Resamples)

| Point | %      | SD    | 95% CL | <u>.(Exp)</u> | Skew    |
|-------|--------|-------|--------|---------------|---------|
| IC25  | 67.549 | 3.218 | 50.594 | 69.585        | -2.2573 |



| Start Date: 10/11/2011 | Test ID: 13888 | Sample ID: | BP\_09/1 005 TV-1 10 | Date: 10/18/2011 | Lab ID: ENVIRON | Sample Type: | EFF2-Industrial | Protocol: EPAFW02-EPA/821/R-02-01 Test Species: | PP-Pimephales promelas | Comments: | Comments: | Comments | Comme

#### **Dose-Response Plot**

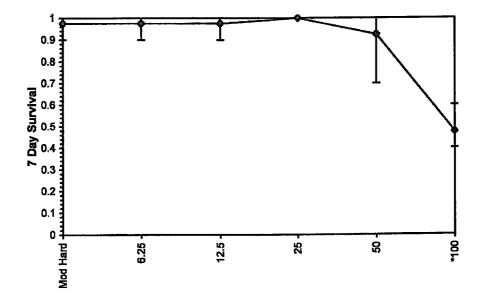


|   |                          |        | La                  | rval Fish Gr     | owth and Surviv | al Test-7 Day Su           | ırvival   |
|---|--------------------------|--------|---------------------|------------------|-----------------|----------------------------|---|
| Start Date:<br>End Date:<br>Sample Date:<br>Comments: | 10/11/2011<br>10/18/2011 |        | Test ID:<br>Lab ID: | 13888<br>ENVIRON | EPA/821/R-02-01 | Sample ID:<br>Sample Type: | BP_901005 או/רוכו ובן בול בו<br>EFF2-Industrial<br>PP-Pimephales promelas |
| Conc-%  | 1                        | 2      | 3                   | 4                |                 |                            |   |
| Mod Hard  | 1.0000                   | 0.9000 | 1.0000              | 1.0000           |                 |                            |   |
| 6.25  | 0.9000                   | 1.0000 | 1.0000              | 1.0000           |                 |                            |   |
| 12.5  | 1.0000                   | 0.9000 | 1.0000              | 1.0000           |                 |                            |   |
| 25  | 1.0000                   | 1.0000 | 1.0000              | 1.0000           |                 |                            |   |
| 50  | 1.0000                   | 1.0000 | 1.0000              | 0.7000           |                 |                            |   |
| 100   | 0.5000                   | 0.4000 | 0.4000              | 0.6000           |                 |                            |   |

|          |        |        | Tra    | ansform: | Arcsin Sc | uare Roo | Rank | 1-Tailed |          |  |
|----------|--------|--------|--------|----------|-----------|----------|------|----------|----------|--|
| Conc-%   | Mean   | N-Mean | Mean   | Min      | Max       | CV%      | N    | Sum      | Critical |  |
| Mod Hard | 0.9750 | 1.0000 | 1.3713 | 1.2490   | 1.4120    | 5.942    | 4    |          |          |  |
| 6.25     | 0.9750 | 1.0000 | 1.3713 | 1.2490   | 1.4120    | 5.942    | 4    | 18.00    | 10.00    |  |
| 12.5     | 0.9750 | 1.0000 | 1.3713 | 1.2490   | 1.4120    | 5.942    | 4    | 18.00    | 10.00    |  |
| 25       | 1.0000 | 1.0256 | 1.4120 | 1.4120   | 1.4120    | 0.000    | 4    | 20.00    | 10.00    |  |
| 50       | 0.9250 | 0.9487 | 1.3068 | 0.9912   | 1.4120    | 16.103   | 4    | 17.50    | 10.00    |  |
| *100     | 0.4750 | 0.4872 | 0.7602 | 0.6847   | 0.8861    | 12.679   | 4    | 10.00    | 10.00    |  |

| Auxiliary Tests                   |              |            |                |    | Statistic | Critical | Skew    | Kurt    |
|-----------------------------------|--------------|------------|----------------|----|-----------|----------|---------|---------|
| Shapiro-Wilk's Test indicates nor | n-normal dis | stribution | $(p \le 0.05)$ |    | 0.83563   | 0.916    | -1.6008 | 3.51162 |
| Equality of variance cannot be co |              |            |                |    |           |          |         |         |
| Hypothesis Test (1-tail, 0.05)    | NOEC         | LOEC       | ChV            | TU |           |          |         |         |
| Steel's Many-One Rank Test        | 50           | 100        | 70.7107        | 2  |           |          |         |         |
| Treatments vs Mod Hard            |              |            |                |    |           |          |         |         |

#### Dose-Response Plot



#### ENVIRON FATHEAD MINNOW SURVIVAL AND GROWTH 7-DAY CHRONIC TOXICITY TEST EPA-821-R-02-013 Method 1000.0

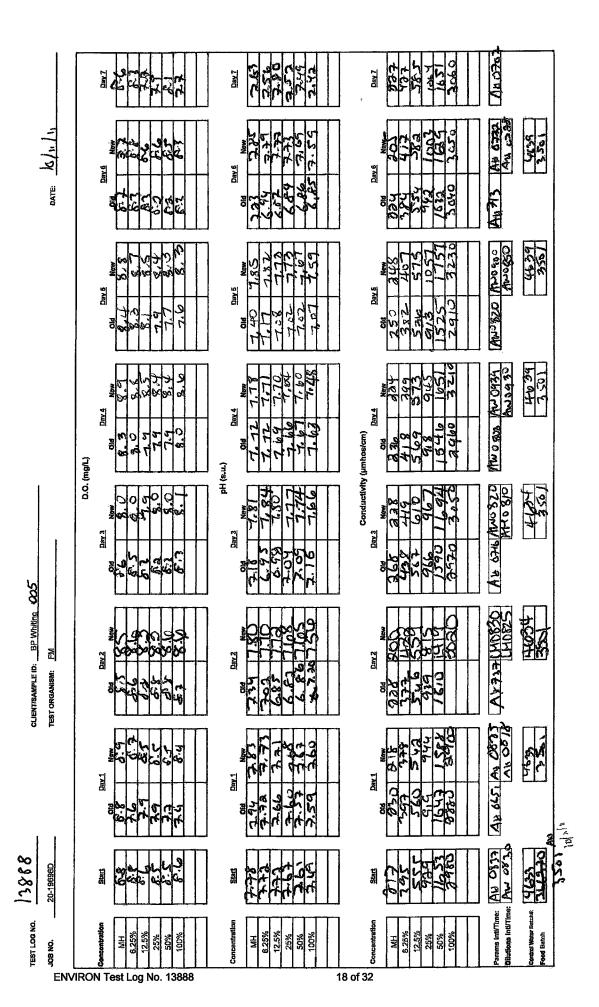
| TEST LOG NO.:  JOB NUMBER:: 20-19696D  INDUSTRY: BP Whiting  EFFLUENT: 005  DILUTION WATER: Mod Hard  NPDES: Yes No 1  FOOD BATCH: 100 | BEGINNING: HRS: 103° DATE: 10 18 11  ENDING: HRS: 123° DATE: 10 18 11  TEST DILUTIONS: 6.25 - 100  ORGANISM AGE (date): 10 10 11  ORGANISM SOURCE: ECT # 36 2 3  SOURCE TEMP @ TEST START: 24.0°C  RANDOMIZED BY: | PHOTOPERIOD: 16 hr light/6 hr dark FEEDING REGIME: 0.15 mL Artemia @ 2 times/day TEST VESSEL CAPACITY: 450 mL TEST SOLUTION VOLUME: 250 - 300 mL NO, ORGANISMS/TREATMENT: 10 NO, REPLICATES: 4 |
|--|---|--|
|--|---|--|

|                   |                  |          |          |                | SURVI           | VAL (#)       |                |   |           |
|-------------------|------------------|----------|----------|----------------|-----------------|---------------|----------------|---|-----------|
| CONC<br>(%)       | REP<br>ID        | START    | DAY 1    | DAY 2          | DAY 3           | DAY 4         | DAY 5          | DAY 6   | DAY 7     |
| Mod Hard          | Α                | 10       | ( O      | 10             | 10              | 10            | 10             | 70  | 10        |
|                   | В                | 10       | lą       | 10             | 4               | 9             | 9              | 9   | ٩         |
|                   | С                | (0       | 13       | 10             | 10              | 10            | 10             | 61  | 10        |
|                   | D                | 10       | l)       | 10             | 10              | 10            |                | 10  | 10        |
|                   | E                |          |          |                |                 |               |                |   |           |
|                   | Temp(°c):old/new | 24.4     | 24.21040 | OUG IOUG       | MOIDE           | Supplied.     | aus love       | 24.9 4.2                                      |           |
| 6.25%             | Α                | 10       | 10       | a              | d               | 9             | 9              | 9   | 9         |
|                   | В                | 10       | U        | 10             | 10              | 10            | 10             | 10  | 10        |
|                   | С                | ΙO       | lo       | 10             | 10              | 10            | 10             | <u>  10</u>                                   | 10        |
|                   | D                | 10       | la       | 10             | 10              | 10            | 10             | 13  | 10        |
|                   | E                |          | ,        |                |                 |               |                |   |           |
|                   | Temp(°c):old/new | 24.3     |          | <b>BUDIBUS</b> | DUDGUC          | DADI940       |                | 313/21%                                       | 24:5)     |
| 12.5%             | A                | 10       | lo       | 10             | 10              | 10            | 10             | (2)   | 10        |
|                   | В                | 10       | 13       | 10             | q               | 9             | 9              | q   | 9         |
|                   | C                | 10       | 10       | 10             | 10              | 10            | 10             | 10  | 10        |
|                   | D                | 10       | 9        | 10             | 10              | 10            | 10             | 10  | 10        |
|                   | E                |          |          |                |                 |               |                |   |           |
|                   | Temp(°c):old/new | 24.4     | 14.0/220 | <u>ouglive</u> | <b>LINGSING</b> | JAC 9141      |                | 24.1/24.1                                     | 2411      |
| 25%               | Α                | 16       | 10       | 10             | 10              | 10_           | 10             | lo_   | 10        |
|                   | В                | (0       | lo       | 10             | 10              | LD            | 10             | 10  | 0         |
|                   | С                | 10       | 10       | 10 _           | <u> 10 </u>     | 10            | 10             | اه  | 0)        |
|                   | D                | 10       | 10       | l lō           | 10              | 10            | 10             | <u>                                      </u> | 10        |
|                   | E                |          |          |                |                 |               |                |   |           |
|                   | Temp("c):old/new | 24.7     | 24.0/240 | SUSTON         | とういろ            | MAGNO         | Moldio         | 347 341                                       | 24.9      |
| 50%               | Α                | 10       | 10       | 10             | 10              | 10            | <u> </u>       | <u> </u>                                      | 0         |
|                   | В                | 0        | (0       | 10             | 10              | 10            | 10             | 10  | 10        |
|                   | С                | 10       | 13       | lo             | 10              | 10            | 10             | 10  | 10        |
|                   | ם                | 10       | 10       | 10             | 10              | 9             | 9              | +   | 7         |
|                   | E                |          |          |                |                 |               |                |   |           |
|                   | Temp(°c):old/new | 245      | 24. 1840 | UN OJANO       |                 |               | <b>DIO1910</b> | 1 '1 3  | 25,1      |
| 100%              | Α                | 16       | 10       | 10             | 10              | 9             | 7              | 6   | 5         |
|                   | В                | 01       | 10       | 10_            | 9               | 9             | 7              |   | 4         |
|                   | С                | ια       | 16       | ال ا           | 9               | 8             | 8              | <del></del>                                   | 4         |
|                   | D                | 10       | (0       | 9              | <u>  †</u>      | 13-           | <u>+</u>       | 1   | 6         |
| <u> </u>          | E                |          |          |                |                 | AL 100 E      |                | AE TAU  | 7/(0      |
|                   | Temp(°c):old/new | 24.7     |          |                |                 | <b>SIDMAG</b> |                | 341 24.9                                      | 24.8      |
| Test Renewal      | Time             | 1630     | 1433     | 1034           | 0935            | Mya           | 1154           | 094a  | 10 [18])) |
|                   | Date<br>Initials | 10/11/11 | 18/18/19 | 10 13 11<br>HM | 191411          | DISIL         | HM             | AH  | AW        |
| morning feeding   | Int/Time         |          |          | Ln0700         | JAGJAE A        |               | Awa 154        | LMOTUS  |           |
| afternoon feeding | Int/Time         |          | 777177   | HUICUE         |                 |               | AMIS35         | AWIORD  | 12161142  |

# ENVIRON FATHEAD MINNOW SURVIVAL AND GROWTH 7-DAY CHRONIC TOXICITY TEST EPA-821-R-02-013 Method 1000.0

| TEST LOG NO.: 1388 BEGINNING: HRS: 1030 DATE: 10 / 11/1 | PHOTOPERIOD: 16 hr light      |
|---|-------------------------------|
| JOB NO.: 20-19696D ENDING: HRS 20 DATE: 1011811         | FEEDING REGIME:               |
| INDUSTRY: RP Whiting                                    | 0.15 mL Artemia @ 2 times/day |
| EFFLUENT: Out COLL NO. ORGANISMS/TREATMENT: 10          | TEST VESSEL CAPACITY: 450 mL  |
| NPDES: Yes No 005 NO. REPLICATES: 4                     | TEST SOLUTION VOLUME: 250 mL  |
| T(x)  |                               |

|          |     |         |           | GROWTH RE   | SULTS    |          |                        |                      |                  |
|----------|-----|---------|-----------|-------------|----------|----------|------------------------|----------------------|------------------|
| CONC     | REP | Boat ID | Tare wt   | Combined    | Tot Fish | # of     | Fish Wt (mg)           |                      | 1                |
| (%)      | ID  | 63      | (g)       | wt (g)      | wt (g)   | Fish     | Per Final<br># of Fish | HH IOIG              |                  |
| Mod Hard | Α   | 1       | 108769    | 1.099BH     | 0.00015  | 19       | 0.515                  |                      |                  |
|          | В   | 2       | 1.10044   | 1.10499     | 000455   | 4        | 0.505                  | AVG Control          |                  |
|          | С   | 3       | 108000    | 1085AU      | D.0050k  | 13       | D.500                  |                      | μq               |
|          | D   | 4       | 109140    | 110a18      | 0.000049 | 19       | 0549                   | (using final #)      |                  |
|          | E   |         |           |             |          |          |                        | Arkene               |                  |
| 6.25%    | Α   | 5       | 1.11335   | 111781      | BOOK IN  | 9        | 0000                   | le Houses            | 1                |
|          | В   | 6       | 1.11038   | 113134      | 000494   | Įυ       | ]                      | フ                    |                  |
|          | С   | 7       |           | 1.09931     | 1F400.0  | 10       | Oven ID:               |                      |                  |
|          | D   | 8       | 402901    | 1.09511     | 100000t  | ()       | ]                      |                      |                  |
|          | E   |         |           |             |          |          | ]                      |                      |                  |
| 12.5%    | Α   | 9       | 1.00837   | 14441       | 1-capard | 10       | inst                   |                      |                  |
|          | В   | 10      | 168391    | PUGGO:1     | Bustoo   | 9        | Temp 109               | Tins in: 10 19 14 1. | 118              |
|          | C   | 11      | HUPOUCI   | 112401      | PERCO O  | 10       | I                      | Date/fime            | H                |
|          | D   | 12      | 1.100190  | PEFOIL      | D001063  | 10       | Temp 100               | Tins Out: 10 1911    | $\mathbb{I}_{O}$ |
|          | E   |         |           |             |          |          | ]                      | Date/Time            | H                |
| 25%      | Α   | 13      | 1100mg    | 1.11539     | D00597   | 10       |                        |                      |                  |
|          | В   | 14      | 1.029D9   | 1.09599     | 0.00690  | ΙÚ       | ]                      | FINAL WEIGHTS        | 1                |
|          | C   | 15      | BUGFU-1   | 164838      | 0.00574  | 10       | ]                      | - hal                | 1                |
|          | D   | 16      | 1.10098   | 110195      | FRECOR   | 10       |                        | DATE: 10191          | 1                |
|          | E   |         |           |             |          |          | ]                      | INITIALS: +HM        | 1                |
| 50%      | Α   | 17      | こうかいる     | UPRPOI      | DEDOGOLO | 10       | ]                      |                      |                  |
|          | В   | 18      | LOSIRS    | 1.00817     | 0.00033  | 10       | 1                      |                      | H                |
| J        | С   | 19      | 100200    | 1.08813     | 40E000   | 10       | ]                      |                      | H                |
|          | D   | 20      | 10000     | IMILES      | 0.0578   | 7        | ]                      |                      |                  |
|          | E   |         | FORM Y    |             |          |          |                        |                      |                  |
| 100%     | A   | 21      | 1.10394   | 1.10439     | 100145   | 5        | 7                      |                      |                  |
|          | В   | 22      |           | CV5V3       | 0.00130  | 4        | 7                      |                      |                  |
|          | C   | 23      | 158756    |             | D.00165  | 4        | ]                      |                      |                  |
|          | D   | 24      |           |             | 0.00308  | 6        | ]                      |                      | H                |
|          | E   |         |           |             |          |          | ]                      |                      |                  |
|          | A   |         |           |             |          |          | 7                      |                      | 1                |
|          | В   |         | , <u></u> |             |          | <u> </u> | 7                      |                      | 1                |
|          | C   |         |           | <del></del> |          |          | ]                      |                      | 1                |
|          | D   |         |           |             |          |          | ]                      |                      | 1                |
|          | E   |         |           |             |          |          | ]                      |                      |                  |
|          |     | / Date: | MOIN      | UMINIO      | X        |          | 1 .                    |                      | 1                |



19 of 32

| TEST LOG NO. | 13888     | CLIENT:      | BP Whiting |            | DATE OF TEST: | 10/11/2 |
|--------------|-----------|--------------|------------|------------|---------------|---------|
| JOB NO.      | 20-19696D | TEST TYPE(S) | PERFORMED: | Fm Chronic |               |         |

| 100% EFFLUEN | NT<br>Sample T <sup>IH 12</sup> | √¹ <sup> ¹¹</sup> Sample | 1st Use   | Hardness   | Alkalinity | TRC  | NH₃N  |
|--------------|---------------------------------|--------------------------|-----------|------------|------------|------|-------|
| Batch #      | ID 005                          | Date                     | Dațe      | mg/L CaCO3 | mg/L       | mg/L | mg/L  |
| 14195        | Outfallson                      | 10/9-10/11               | 10/11/11  | aa 8       | 160        | 0.09 | 20.1  |
| 14207        | Or76911000                      | 11/21-11/01              | 10/13/11  | <u> </u>   | 100        | 0.63 | 40.1  |
| 14720        | Outfaller 095                   | ाठोष्ड-१पी।              | 10  15/11 | वावे       | 35         | 0.08 | 0.132 |
|              |                                 |                          |           |            |            |      |       |
|              |                                 |                          |           |            |            |      |       |
|              |                                 |                          |           |            |            |      |       |

#### **CONTROL / DILUTION WATER**

| Batch # | Sample<br>ID | Sample<br>Date | 1st Use<br>Date | Hardness<br>mg/L CaCO3 | Alkalinity<br>mg/L | TRC<br>mg/L |
|---------|--------------|----------------|-----------------|------------------------|--------------------|-------------|
| 4633    | MH           | 1015[[1        | 199111          | 84                     | 51                 | <0.0.9      |
| 4634    | MH           | 105111         | IDUALLI         | 864                    | 52                 | <0.09       |
| 4634    | MH           | 10/9/11        | Plidi           | 85.0                   | 50_                | 6007        |
|         |              |                |                 |                        |                    |             |
|         |              |                |                 |                        |                    |             |

Attachment 4: Chain-of-Custody Forms

20-19696D € N V I R O N

CUSTOF! SEAT



| E<br>N | roject Name:                               |             | الم سيسر مي       | Project Number                  | or:             | / D              |        | ľ                      | L                    |                        | Ana                      | lysis               | Re                     | ques                              | ted                    |                      |                       |                      |                              |
|--------|--|-------------|-------------------|---------------------------------|-----------------|------------------|--------|------------------------|----------------------|------------------------|--------------------------|---------------------|------------------------|-----------------------------------|------------------------|----------------------|-----------------------|----------------------|------------------------------|
|        | Project Name: BP W<br>OOI BP W<br>ndustry: | ETT         | es ling           | 30-                             | 19676           |                  |        | -                      |                      |                        |                          |                     |                        |                                   |                        |                      | ,<br>NG               | CHAIN-O              | F-CUSTODY                    |
| ᆌ      | Phone: 9-473-                              |             | FAX:              |                                 |                 |                  |        | 1                      |                      |                        |                          |                     |                        | 20                                |                        |                      | B                     |                      |                              |
| 3      | 319-713-<br>County: 1 3 c p                | 3/ <u>a</u> | City:             | 1./                             |                 | Ştate:           |        | 1                      | >                    | L                      | ubia                     | . ]                 | 8                      | dubi                              | ts                     |                      | K                     | ENV                  | IRON                         |
| J      | レタハモ                                       | 95          | .WY               | iting                           | INPDES Pe       | bana             |        | ters                   | ino                  | shine                  | nia d                    | rjex                |                        | hrita                             | Tes                    | sts                  | 11/2                  |                      | w Drive, Suite 300           |
| 8      | Sample Collected by (print<br>Terry C.     | aV 5        |                   | _                               |                 | ,                |        | Total Volume in liters | Acute Fathead minnow | Acute Bannerfin shiner | Acute Ceriodaphnia dubia | Acute Daphnia pulex | Chronic Fathead minnow | Chronic <i>Ceriodaphnia dubia</i> | Continuous Batch Tests | Discrete Batch Tests | oxicitu               | Brentwoo             | d, TN 37027<br>115) 377-4775 |
|        | Sample Collected by (sign                  |             |                   |                                 | NPDES Te        | st:<br>Yes       | No.    | olem<br>m              | Fathe                | Bann                   | Ceric                    | Dapt                | c Fat                  | 9<br>2                            | snon                   | te Ba                |                       | FAX: (61             | 5) 377-4976                  |
|        | 1 my                                       | T "         | Mez               |                                 |                 |                  | of     | otal                   | cute                 | cute                   | cute                     | cute                | hron                   | hron                              | ontin                  | iscre                | Other                 | Description          |                              |
| ı      | Sample Location / ID                       | Comp/Grab   | Container<br>Type | Chilled During Collection (Y/N) | Start Date/Time | End<br>Date/Time | Cntrs  | <del>  -</del>         | A                    | A                      | A                        | ⋖                   | 0                      | ٥                                 | 0                      |                      | 0                     | Definitive or Screen | Sample B# (lab only)         |
|        | OUTFALL DOT                                | COMP        | IOL               | У                               | 18-9-11         | 10-19-11         | T      | 7.0                    | =1                   |                        |                          |                     |                        |                                   |                        |                      | X                     |                      | 14195                        |
|        | 005  |             |                   |                                 |                 |                  |        |                        |                      |                        |                          |                     |                        |                                   |                        |                      |                       |                      |                              |
| 3      | Tet  | 12/1/11     |                   |                                 |                 |                  |        | ļ                      |                      |                        |                          |                     |                        |                                   | -                      |                      |                       |                      |                              |
| 의<br>강 |  | <u> </u>    |                   |                                 |                 |                  |        | -                      |                      |                        |                          |                     | -                      |                                   |                        |                      |                       |                      | 1-2 (4.25,ex)                |
|        |  |             |                   |                                 |                 |                  |        |                        |                      |                        |                          |                     |                        | $\dashv$                          |                        |                      | 4                     |                      |                              |
|        |  |             |                   | •                               |                 |                  |        |                        |                      |                        |                          |                     |                        |                                   |                        |                      |                       |                      |                              |
|        |  |             |                   |                                 |                 |                  |        |                        |                      |                        |                          |                     |                        |                                   |                        |                      |                       |                      |                              |
|        | Matrix: SS - Soli GW - Groun<br>Remarks:   | ndwater W   | - Wastewater      | AW - Amblent Wa                 | ter ML - Mixe   | d Liquor SL -    | Sludge | SD - S                 | edime                | nt O                   | T - Ot                   | her_                |                        |                                   |                        |                      |                       |                      |                              |
|        | Measured TRC (if applica                   | ble):       |                   | _mg/L                           |                 |                  |        |                        |                      |                        |                          |                     |                        |                                   |                        |                      |                       |                      |                              |
|        | Relinquished by: (Signature)               | i des       | Date:             | Time: 12:00                     | Received by:    | (Signature)      |        |                        |                      |                        | Samp<br>edEx             |                     | hippe<br>Oth<br>Cou    | er                                |                        | =                    | UPS<br>Hand<br>Delive |                      | (lab use enly)               |
| 7.1    | Relinquished y: (Signature)                |             | Date:             | Time:                           | Received by:    | (Signature)      |        |                        |                      | Rece                   | (pt Te                   | SHELLING THE        | 330                    |                                   | Contr                  | alners               |                       | me Received:         | · 李教·龙·                      |
|        | Relinquished by: (Signature)               |             | Date:             | Time:                           | f = f           | lab by: (Signa   |        | <b>*</b>               |                      | Date:                  | 5/4                      | ij,                 | 1                      |                                   | Time                   |                      |                       | PH upon arrival:     | 00 upon arrival:             |

| Sample, Receipt Checklist:  |    |
|---|----|
| Client: Pruhing Sample ID(s) Odfall Dot 005 74                      |    |
| Batch #'s 1467  |    |
| 1. Date/Time received 10/13/11 0776 by A19                          |    |
| 2. Cooler sealed and intact upon arrival? Yes No                    | 0  |
| 3. Custody seals present? Yes No                                    |    |
| 4. Samples received below 6 degrees Celsius? (Yes) No               |    |
| 5. Was ice present? No  |    |
| 6. Is the COC filled out correctly including the sample date/time a | n  |
| signed? Yes No  | _  |
| 7. Was the sample received within 36 hours of collection?           | es |
| No  |    |
| 8. Did the sample(s) arrive in good condition?                      |    |
| No  |    |
| 9. Was pH and DO measured and in range? Yes                         |    |

Comments:

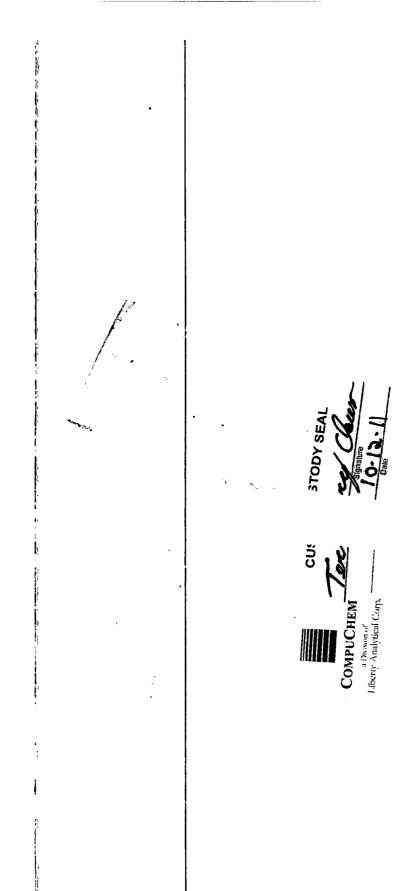
10.

No

No

Was residual chlorine present?

0.03



ple

| M<br>Zn | 300                                      |                |                                       |                  |               | <del></del>                           |             | ,                      |                      |                        | A                        |                     |                        |                                   |                        |  |                       |                                   |  |
|---------|--|----------------|---------------------------------------|------------------|---------------|---------------------------------------|-------------|------------------------|----------------------|------------------------|--------------------------|---------------------|------------------------|-----------------------------------|------------------------|--|-----------------------|-----------------------------------|--|
| SIRON I | Project Name:                            | WE             | T Test                                | Project Number   | 20-1          | 9696                                  | D           |                        |                      |                        | Ana                      | alysis              | He                     | ques                              | tea                    |  | 9                     | CHAIN-O                           | F-CUSTODY  |
| Top.    | Manager y                                |                | FAV.                                  |                  |               | · · · · · · · · · · · · · · · · · · · |             |                        |                      |                        |                          |                     |                        |                                   |                        |  | Test/ng               | •                                 |  |
| 3       | 219-473-3                                | 1a6            | FAX:                                  |                  |               |                                       |             |                        |                      |                        | Įa                       |                     | >                      | ubia                              |                        |  |                       | -                                 |  |
| Л       | County: Lake                             |                | City: Wh.                             | Tina             | 1             | State: /                              | a           | Į g                    | ₹                    | Jer I                  | dub                      | ×                   | OLU                    | nia d                             | ests                   | ø                                      | Ĥ,                    | ENV                               | IRON   |
| 3885    | Sample Collected by (print               | ):<br>1        |                                       | J                | NPDES Per     | rmit No.:                             |             | ו lite                 | 盲                    | n shi                  | shrife                   | pule                | E                      | laphi                             | 뒫                      | Tes                                    | _IJ                   |                                   | w Drive, Suite 300<br>d, TN 37027  |
| L       | Sample Collected by (signature)          | 119VS          | _                                     |                  | NPDES Te      | nte .                                 |             | me i                   | head                 | nerfi                  | toda                     | hnia                | athe                   | erioc                             | is Ba                  | atch                                   | axle                  | PHONE: (6                         | 15) 377-4775<br>5) 377-4976  |
| ľ       | Sample Conscied by (signa                |                | 7<br>                                 |                  | No No         | Yes                                   | No.         | Total Volume in liters | Acute Fathead minnow | Acute Bannerfin shiner | Acute Ceriodaphnia dubia | Acute Daphnia pulex | Chronic Fathead minnow | Chronic <i>Ceriodaphnia dubia</i> | Continuous Batch Tests | Discrete Batch Tests                   | H                     | ·                                 |  |
| -       | Sample Leation / ID                      | Comp/Grab      | Container                             | Chilled During   | Start         | End                                   | of<br>Cntrs | Total                  | Acute                | Acute                  | Acute                    | Acute               | <u>S</u>               | Pro                               | Sont                   | Discr                                  | Other                 | Description  Definitive or Screen | Sample B# (lab only)   |
| ĺ       | ouripio igualion.                        | Jonny, G. a.b. | Туре                                  | Collection (Y/N) | Date/Time     | Date/Time                             | - CALLED    | İ                      |                      |                        |                          |                     |                        | Ť                                 |                        |  |                       | DOM:1010 01 00100.1               | Compage Compage  |
|         | 30TFall 201                              | Comp           | 10 L                                  | У                | 0700          | 10-12-11                              |             | 8, Ô                   |                      |                        |                          |                     |                        |                                   |                        |  | X                     |                                   | 14207  |
|         | 005                                      |                |                                       |                  |               |                                       | Ľ           |                        |                      |                        |                          |                     |                        |                                   |                        |  |                       |                                   |  |
| ᇧ       | 7412/                                    | 1/11           | · · · · · · · · · · · · · · · · · · · |                  |               |                                       |             | <u> </u>               |                      |                        |                          |                     | 4                      |                                   |                        |  | -                     |                                   |  |
| 3       |  |                |                                       |                  |               |                                       |             | ļ                      |                      |                        |                          |                     |                        |                                   |                        |  |                       |                                   | and the state of t |
| ٦,      |  | <u> </u>       |                                       |                  |               |                                       | ļ           | ļ                      |                      |                        |                          |                     |                        |                                   |                        |  |                       |                                   |  |
| ŀ       |  |                |                                       |                  |               | L                                     |             | <u> </u>               |                      |                        |                          |                     |                        |                                   |                        |  |                       |                                   |  |
| ł       |  |                |                                       |                  |               |                                       |             | <del> </del>           |                      |                        |                          |                     |                        |                                   |                        |  |                       |                                   |  |
|         | Matrix: SS - Soll GW - Groun<br>Remarks: | ndwater www    | - Wastewalds                          | AW - Ambient Wa  | ter ML - Mixe | d Liquor SL -                         | Sludge      | SD - S                 | edime                | nt O                   | T - Ot                   | her_                |                        |                                   |                        |  |                       |                                   |  |
|         | Measured TRC (if applicat                | ole):          |                                       | _mg/L            |               |                                       |             |                        |                      |                        |                          |                     |                        |                                   |                        |  |                       | <u> </u>                          |  |
| +       | Relinguished by: (Signature)             | lues           | Date:                                 | Time:     300    | Received by:  | (Signature)                           |             |                        |                      | F                      | Sam;<br>edEx             | ples s              | hippi<br>Oth<br>Cou    | er                                |                        | =                                      | UPS<br>Hand<br>Delive |                                   | (inb use only)   |
|         | Relinquished by (Signature)              |                | Date:                                 | Time:            | Received by:  | (Signature)                           |             |                        |                      | Rece                   | ipi Te                   | mp:                 |                        | -/2909P                           | Cont<br>(              | C2000000000000000000000000000000000000 | A49-44000A4/7         | me Received:                      | \$ 1400 S  |
|         | Relinquished by: (Signature)             |                | Date:                                 | Time:            |               | lab by: (Signa                        |             |                        |                      | Dale:                  |                          | ( }                 | (,,                    |                                   | Time                   | →?                                     |                       | pH upon arrival:<br>フリン           | 9.2  |

| Sampl | e Rece | ipt Ch | ecklist: |
|-------|--------|--------|----------|
|-------|--------|--------|----------|

Client: Rowhing Sample ID(s) Outfall cost 005 Tul

Batch #'s 14195

- 1. Date/Time received 6/11/11 0745
- 2. Cooler sealed and intact upon arrival?

No

3. Custody seals present?

No No

Yes'

- 4. Samples received below 6 degrees Celsius? Yes
- No

- 5. Was ice present?
- 6. Is the COC filled out correctly including the sample date/time and (Yes) signed? No
- 7. Was the sample received within 36 hours of collection? No
- 8. Did the sample(s) arrive in good condition?



No

9. Was pH and DO measured and in range?



No

Was residual chlorine present? 10. No



Comments:

TRC 0.09

ument ID WRU-DFNV-4G05-44456

BP Whiting
10/15/11



| ENVIR  | Project Name: B P                |  | Analysis Requested      |                                       |                                       |                            |  |  |  | CHAIN-OF-CUSTODY |         |                            |                        |   |            |   |                                  |                               |
|--|----------------------------------|--|-------------------------|---------------------------------------|---------------------------------------|----------------------------|--|--|--|------------------|---------|----------------------------|------------------------|---|------------|---|----------------------------------|-------------------------------|
| ON Tes   | Industry:                        | •  |                         | · · · · · · · · · · · · · · · · · · · | <i></i>                               |                            |  |  |  |                  |         |                            |                        |   |            | 1,2                                     | CHAIN-C                          | P-COSTODY                     |
| ENVIRON Test Log No. 13888   | County:                          | 114-7)3-3/d6<br>nty: / 1. City: . 1/-1State! |                         |                                       |                                       |                            | \\\/\/\/\/\/\/\/\\\\\\\\\\\\\\\\\\\\\\ |  |  |                  |         |                            |                        | dubia   |            | 1/2/2                                   | ENV                              | IRON                          |
| 13888  | Sample Collected by (print)      | NPDES Permit No.:                            |                         |                                       | in liters                             | I minno                    | in shine                               | Ceriodaphnia dubla                                       | xejnd a  | ad min           | daphnia | Discrete Batch Tests       | -5                     | 201 Summit View Drive, Suite 300<br>Brentwood, TN 37027 |            |   |                                  |                               |
|  | Sample Collected by (signature)  | ature):                                      | 1                       |                                       | NPDES Te                              | st:                        | No.                                    | Total Volume in liters                                   | Volume in liters Fathead minnow Bannerfin shiner |                  |         | Acute <i>Daphnia pulex</i> | Chronic Fathead minnow | Chronic <i>Ceriodaphnia di</i>                          | te Batch   | 76X                                     | PHONE: (6                        | 615) 377-4775<br>15) 377-4976 |
|  | Sample Location / ID             | Comp/Grab                                    | Container               | Chilled During                        | Start                                 | End                        | of<br>Cntrs                            | Total \  | Acute  | Acute            | Acute   | Acute                      | Chron                  | Chroni  | Discre     | Other-                                  | Description Definitive or Screen | Sample B# (lab only)          |
|  |                                  |  | Type                    | Collection (Y/N)                      | Date/Time                             | Date/Time                  |  |  |  |                  |         |                            |                        |   |            |   |                                  | Sales Land                    |
|  | OUTFELL DOT                      | Comp   | 10 L                    | <u> </u>                              | 0700                                  | 18-18-01                   |  | 9.0  |  |                  |         |                            |                        |   |            | X                                       |                                  | 14220                         |
|  | 005<br>TUH 121                   | 7/11   |                         |                                       | · · · · · · · · · · · · · · · · · · · |                            | <u> </u>                               |  |  |                  |         |                            |                        | _   |            |   |                                  |                               |
| 28   | 1011101                          | 777  |                         | -                                     |                                       |                            |  |  |  |                  |         |                            |                        | -   |            |   |                                  | 200                           |
| of 32  | <del> </del>                     |  |                         |                                       |                                       |                            | <del> </del>                           |  |  | -                |         |                            |                        |   |            |   |                                  |                               |
|  |                                  |  |                         |                                       |                                       |                            |  | ļ  |  |                  |         |                            |                        |   |            |   |                                  | Appendix and                  |
|  |                                  |  |                         |                                       | -                                     |                            |  |  |  |                  |         |                            |                        |   |            |   |                                  |                               |
| ;  |                                  |  |                         |                                       |                                       |                            |  |  |  |                  |         |                            |                        |   |            |   |                                  | - 23                          |
| * Matrix: SS - Soil GW - Groundwater WW - Wastewater WW - Ambient Water ML - Mixed Liquor SL - Sludge SD - Sediment OT - Other |                                  |  |                         |                                       |                                       |                            |  |  |  |                  |         |                            |                        |   |            |   |                                  |                               |
|  | easured TRC (if applicable):mg/L |  |                         |                                       |                                       |                            |  |  |  |                  |         |                            |                        |   |            |   |                                  |                               |
|  | Relinquished by: (Signature)     | Received by:                                 | eceived by: (Signature) |                                       |                                       |                            |  | Samples shipped via: UPS FedEx Other Hand Courier Delive |  |                  |         |                            |                        | Condition: (lab use only)                               |            |   |                                  |                               |
|  | Relinquished (Signature)         |  | 10-14-11<br>Date:       | 12:00<br>Time:                        | Received by:                          | (Signature)                | .·                                     |  |  | Rece             | lpt Te  | mp:<br>⊃.·l                | ي د                    | Car   | Again anns | Activistic States                       | ma Received:                     |                               |
|  | Relinquished by: (Signature)     |  | Date:                   | Time:                                 | Received for                          | iab by: (Signa<br>ha Payon | (enut)<br>ノーし                          | الله المراد  | to-  | Date:            | ol.     | ادا                        | 1                      | Tin<br>(  | 847<br>847 | 111111111111111111111111111111111111111 | pH upon arrival:                 | DO upon arrival:              |
|  |                                  |  |                         | · · · · · · · · · · · · · · · · · · · |                                       | J                          |  |  |  |                  | -       | 97                         |                        |   |            |   |                                  |                               |

| Sample, Receipt Checklist:  |  |  |  |  |  |  |  |  |  |  |
|---|--|--|--|--|--|--|--|--|--|--|
| Client: BP Whiting Sample ID(s) Dutfall Got The 11/1                  |  |  |  |  |  |  |  |  |  |  |
| Batch #'s 14226   |  |  |  |  |  |  |  |  |  |  |
| 1. Date/Time received 10 15/11 0842 by AW                             |  |  |  |  |  |  |  |  |  |  |
| 2. Cooler sealed and intact upon arrival? No                          |  |  |  |  |  |  |  |  |  |  |
| 3. Custody seals present? Yes No                                      |  |  |  |  |  |  |  |  |  |  |
| 4. Samples received below 6 degrees Celsius? Yes No                   |  |  |  |  |  |  |  |  |  |  |
| 5. Was ice present? No  |  |  |  |  |  |  |  |  |  |  |
| 6. Is the COC filled out correctly including the sample date/time and |  |  |  |  |  |  |  |  |  |  |
| signed? Yes No  |  |  |  |  |  |  |  |  |  |  |
| 7. Was the sample received within 36 hours of collection? Yes         |  |  |  |  |  |  |  |  |  |  |
| No  |  |  |  |  |  |  |  |  |  |  |
| 8. Did the sample(s) arrive in good condition? Yes                    |  |  |  |  |  |  |  |  |  |  |
| No  |  |  |  |  |  |  |  |  |  |  |
| 9. Was pH and DO measured and in range? Yes                           |  |  |  |  |  |  |  |  |  |  |
| No  |  |  |  |  |  |  |  |  |  |  |
| 10. Was residual chlorine present? Yes                                |  |  |  |  |  |  |  |  |  |  |
| No TRC 0.08   |  |  |  |  |  |  |  |  |  |  |

Comments:

Attachment 5: Reference Toxicant Data

20-19696D

ENVIRON

#### Fathead Minnow CHRONIC REFERENCE TOXICANT TESTING-SODIUM CHLORIDE (NaCI) 2010 - 2011

|                | Log<br>Number | Test<br>Initiation<br>Date | Control<br>Survival<br>(%) (*) | Control Mean<br>Dry Weight<br>(mg/fish) (*) | SURVIVAL       |                | GRÓWTH         |                |             | IC25            | IC25<br>CUMULATIVE | IC25               | IC25            | IC25            | Coefficient         |
|----------------|---------------|----------------------------|--------------------------------|---|----------------|----------------|----------------|----------------|-------------|-----------------|--------------------|--------------------|-----------------|-----------------|---------------------|
| Test<br>Number |               |                            |                                |   | NOEC<br>(mg/L) | LOEC<br>(mg/L) | NOEC<br>(mg/L) | LOEC<br>(mg/L) | PMSD<br>(%) | VALUE<br>(mg/L) | MEAN<br>(mg/L)     | ST. DEV.<br>(mg/L) | 2+<br>STD. DEV. | 2-<br>STD. DEV. | of Variation<br>(%) |
| 1              | 11662         | 04-May-10                  | 97.5                           | 0.246                                       | 6,000          | >6,000         | 1,500          | 3,000          | 28.9        | 2,392           | 2,392              |                    |                 |                 |                     |
| 2              | 12765         | 22-Jun-10                  | 100                            | 0.452                                       | 1,500          | 3,000          | 750            | 1,500          | 12.1        | 1,904           | 2,148              | 345                | 2,838           | 1,458           | 11                  |
| 3              | 12787         | 08-Jul-10                  | 100                            | 0.523                                       | 1,500          | 3,000          | 1,500          | 3,000          | 16.4        | 3,132           | 2,476              | 618                | 3,713           | 1,239           | 20                  |
| 4              | 12824         | 03-Aug-10                  | 95                             | 0.509                                       | 6,000          | >6,000         | 375            | 750            | 16.7        | 934             | 2,091              | 922                | 3,934           | 247             | 38                  |
| 5              | 12870         | 17-Aug-10                  | 100                            | 0.438                                       | 750            | 1,500          | 750            | 1,500          | 20.8        | 1,492           | 1,971              | 842                | 3,654           | 287             | 38                  |
| 6              | 12878         | 20-Aug-10                  | 100                            | 0.433                                       | 750            | 1,500          | 750            | 1,500          | 25.2        | 1,187           | 1,840              | 818                | 3,476           | 204             | 41                  |
| 7              | 12904         | 08-Sep-10                  | 95                             | 0.492                                       | 750            | 1,500          | 750            | 1,500          | 14.6        | 1,124           | . 1,738            | 794                | 3,327           | 149             | 42                  |
| 8              | 12963         | 05-Oct-10                  | 100                            | 0.663                                       | 750            | 1,500          | 750            | 1,500          | 28.7        | 977             | 1,643              | 783                | 3,209           | 77              | 45                  |
| 9              | 13035         | 12-Nov-10                  | 97.5                           | 0.364                                       | 750            | 1,500          | 750            | 1,500          | 22.3        | 1,382           | 1,614              | 738                | 3,089           | 138             | 43                  |
| 10             | 13067         | 07-Dec-10                  | 95                             | 0.445                                       | 750            | 1,500          | 750            | 1,500          | 38.7        | 1,086           | 1,561              | 715                | 2,991           | 131             | 43                  |
| 11             | 13099         | 05-Jan-11                  | 100                            | 0.511                                       | 750            | 1,500          | 750            | 1,500          | 19.7        | 1,286           | 1,536              | 684                | 2,903           | 169             | 42                  |
| 12             | 13147         | 01-Feb-11                  | 100                            | 0.547                                       | 1,500          | 3,000          | 1,500          | 3,000          | 31.2        | 1,737           | 1,553              | 654                | 2,861           | 244             | 40                  |
| 13             | 13214         | 01-Mar-11                  | 95                             | 0.583                                       | 6,000          | >6,000         | 750            | 1,500          | 25.7        | 1,203           | 1,526              | 634                | 2,794           | 258             | 40                  |
| 14             | 13572         | 05-Apr-11                  | 100                            | 0.530                                       | 750            | 1,500          | 760            | 1,500          | 24.2        | 1,106           | 1,496              | 619                | 2,734           | 257             | 40                  |
| 15             | 13648         | 17-May-11                  | 95                             | 0.586                                       | 750            | 1,500          | 750            | 1,500          | 17.7        | 1,140           | 1,472              | 604                | 2,680           | 265             | 40                  |
| 16             | 13666         | 01-Jun-11                  | 97.5                           | 0.680                                       | 1,500          | 3,000          | 750            | 1,500          | 24.5        | 1,246           | 1,458              | 586                | 2,630           | 286             | 39                  |
| 17             | 13725         | 06-Jul-11                  | 100                            | 0.565                                       | 750            | 1,500          | 750            | 1,500          | 26.5        | 1,069           | 1,435              | 575                | 2,586           | 285             | 39                  |
| 18             | 13775         | 02-Aug-11                  | 96                             | 0.534                                       | 1,500          | 3,000          | 1,500          | 3,000          | 10.5        | 2,243           | 1,480              | 590                | 2,659           | 301             | 39                  |
| 19             | 13828         | 07-Sep-11                  | 97.5                           | 0.571                                       | 3,000          | 6,000          | 750            | 1,500          | 17.4        | 1,306           | 1,471              | 574                | 2,620           | 322             | 38                  |
| 20             | 13877         | 04-Oct-11                  | 100                            | 0.579                                       | 1,500          | 3,000          | 750            | 1,500          | 20.4        | 1,385           | 1,467              | 559                | 2,585           | 348             | 37                  |
|                |               |                            |                                |   |                |                |                |                |             |                 |                    |                    |                 | ·               | ·                   |
|                |               | Avg                        | 98                             | 0.513                                       | 1875           | 1950           | 881            | 1763           |             | 1467            | 1718               | 666                | 3015            | 351             | Ī                   |

#### Notes:

Dilution series - 0.375 g/L - 6.0 g/L

NOEC - No Observable Effect Concentration (survival or growth)

LOEC - Lowest Observable Effect Concentration (survival or growth)

ACCEPTABLE TEST RESULTS - A growth NOEC ranging from 750 mg/L to 3,000 mg/L.

(\*) Minimum USEPA CONTROL CRITERIA - 80 percent survival and average dry weight of 0.25 mg (weight based on surviving number of fish).

### **CHRONIC REFERENCE TOXICANT TEST (NaCI) 2010 - 2011 FATHEAD MINNOWS**

